

FILTRATION OF WATER-SEDIMENT SAMPLES FOR THE DETERMINATION OF ORGANIC COMPOUNDS

U.S. GEOLOGICAL SURVEY

Water-Resources Investigations Report 95-4105

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By Mark W. Sandstrom

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CONVERSION FACTORS AND ABBREVIATED WATER-QUALITY UNITS

<u>Multiply</u>	<u>By</u>	<u>To obtain</u>
centimeter (cm)	3.94×10^{-1}	inch
liter (L)	2.64×10^{-1}	gallon
micrometer (μm)	3.94×10^{-5}	inch
milliliter (mL)	2.64×10^{-4}	gallon
millimeter (mm)	3.94×10^{-2}	inch

Degree Celsius ($^{\circ}\text{C}$) may be converted to degree Fahrenheit ($^{\circ}\text{F}$) by using the following equation:

$$^{\circ}\text{F} = 9/5 (^{\circ}\text{C}) + 32.$$

Abbreviated water-quality unit used in this report is as follows:

mL/min milliliter per minute

Other abbreviations also used in this report:

A	ampere
dc	direct current
GC/MS	gas chromatography/mass spectrometry
FEP	fluorinated ethylene propylene
PFA	Teflon-PFA (perfluoroalkoxy)
PVDF	polyvinylidene fluoride
SIM	selected-ion monitoring
SPE	solid-phase extraction
TFE	polytetrafluoroethylene

Filtration of Water-Sediment Samples for the Determination of Organic Compounds

By Mark W. Sandstrom

Abstract

This report describes the equipment and procedures used for on-site filtration of surface-water and ground-water samples for determination of organic compounds. Glass-fiber filters and a positive displacement pumping system are suitable for processing most samples for organic analyses. An optional system that uses disposable in-line membrane filters is suitable for a specific gas chromatography/mass spectrometry, selected-ion monitoring analytical method for determination of organonitrogen herbicides. General procedures to minimize contamination of the samples include preparing a clean workspace at the site, selecting appropriate sample-collection materials, and cleaning the equipment with detergent, tap water, and methanol.

INTRODUCTION

As part of its basic responsibility for the determination of the Nation's water quality, the U.S. Geological Survey needs a nationally consistent procedure for on-site filtration of water samples collected for determination of anthropogenic organic compounds (pesticides, polychlorinated biphenyls, polyaromatic hydrocarbons, phthalate esters, and phenols). Previous reports have described laboratory procedures that are not appropriate for on-site use (Wershaw and others, 1987), or were not suitable for specific compounds, as for example, pesticides (Ward and Harr, 1990). Filtration is useful in studies of the partitioning of organic compounds, where separate analysis of the filtered and sediment-particle-associated phases is needed. Occasionally, large volumes (5 to 40 L) of water are required for sample processing for determination of low concentrations of organic compounds (Foster and others, 1991; Foster and others, 1993), and on-site processing avoids shipping large sample volumes to the laboratory. On-site filtration helps

preserve the integrity of the sample during shipment to the laboratory because filtration removes micro-organisms that can alter the sample. Finally, solid-phase extraction (SPE) procedures for organic compounds require filtration to remove sediment that might block the SPE columns (Sandstrom and others, 1992; Zaugg and others, 1995).

Purpose and Scope

The purpose of this report is to document and describe the equipment and procedures used by the U.S. Geological Survey to filter water-sediment¹ samples from surface and ground water for the determination of anthropogenic organic compounds. More general procedures for the processing of surface-water samples for determination of nutrients, inorganic constituents, and dissolved organic

¹The term "water-sediment" refers to water or a mixture of water and suspended sediment (Ward and Harr, 1990).

carbon are described by Ward and Harr (1990), and this manual complements that publication. Detailed discussion of on-site methods and equipment for measuring fluvial sediment is given by Edwards and Glysson (1988).

Principles and Concepts

Water-sediment samples collected for the determination of organic compounds are filtered to provide a better understanding of the environmental fate of those compounds and to reduce analytical processing errors. Anthropogenic organic compounds (such as organochlorine pesticides, polychlorinated biphenyls, and polyaromatic hydrocarbons) in surface water interact with organic matter attached to sediment particles (particulate organic matter, lipids of organisms) through sorptive processes (Smith and others, 1988). The fate and transport of these compounds need to be considered in both the dissolved and sediment-particle sorbed phases because of these sorptive interactions. Such studies require separate analyses of the organic compounds in filtered and sediment-particle associated phases. In addition, water-sediment mixtures can cause problems during extraction of the organic compounds. In liquid-liquid extraction methods, formation of emulsions during separation of the water and organic solvent phases can cause low recoveries of organic compounds. Also, extraction efficiency for hydrophilic organic compounds can be lower in samples with high concentrations of suspended particles because of inefficient interaction of the particles with the organic solvent. In solid-phase extraction methods that use bonded silica sorbents packed into an extraction column, sediment particles can block the column.

Constituents passing through a filter are operationally defined as "dissolved." Separation of dissolved and particulate material by filtration requires precise definition of the filter medium and

filtration procedure because some material that is not dissolved, for example, colloids, may not be removed by filtration. Dissolved refers to constituents that exist in true chemical solution in a water sample. As a convenient operational definition, the term dissolved is commonly used to refer to constituents in a water sample that has been passed through a filter. Traditionally, the 0.45- μm pore-sized membrane filter has been used to distinguish between dissolved and suspended material because this size filter was the smallest that could be reliably manufactured at the time (Ward and Harr, 1990). The membrane filters commonly used for determination of nutrients and inorganic constituents in water are made of cellulose or polycarbonate polymers. These materials are not suitable for determination of organic compounds because they are not resistant to the organic solvents used to preclean sampling equipment and supplies, or might leach organic contaminants.

Instead, depth filters made of glass fibers are used for determination of organic compounds because they can be precleaned with organic solvents or by baking at 450°C. Depth filters also have a higher loading capacity than membrane filters. Hence they are more suitable for organic sample-preparation methods that frequently require larger sample volumes (1-5 L) than inorganic or nutrient analyses. The 0.7- μm nominal pore-size glass-fiber filter is the smallest size available, and is used operationally to define the "dissolved" and sediment-particle phases of organic compounds. To avoid confusion with the term dissolved, the term filtered will be used in this report to refer to organic compounds in a water sample that has been passed through a 0.7- μm nominal pore-size glass-fiber filter.

Membrane filters made of silver with a 0.45- μm pore size are used to process

water-sediment samples for the determination of dissolved organic constituents (Wershaw and others, 1987) or dissolved organic carbon (Ward and Harr, 1990). Although resistant to organic solvents, these membrane filters become blocked too quickly for their use in filtration of the relatively large sample volumes (1-5 L) required for determination of most organic compounds.

Although plastic membrane filters generally should not be used to process water-sediment samples for the determination of organic compounds, they have been approved for use in at least one specific method. Water-sediment samples analyzed for organonitrogen herbicides by solid-phase extraction and gas chromatography/mass spectrometry (GC/MS) with selected-ion monitoring (SIM) are filtered using nylon membrane filters contained in polypropylene holders (Sandstrom and others, 1992).

EQUIPMENT

Equipment needed for isolation of filtered and suspended-sediment phases of anthropogenic organic compounds by filtration includes glass-fiber filters, disposable membrane filters, aluminum filter units, and positive displacement pumps. These have components made of materials that will not introduce contaminants into the sample and have a low tendency for sorption of organic compounds. These features also make them suitable for use with organic solvents needed in the cleaning procedure.

Although organic compounds generally do not sorb to these materials, special consideration to sample processing might be needed in filtration of dissolved hydrophobic compounds. Adsorption of dissolved or colloidal organic compounds onto glass-fiber filters was demonstrated in a comparison of continuous-flow centrifugation and filtration (Bates and others, 1983).

Glass-Fiber Filters

Application

Binder-free glass-fiber filters with 0.7- μ m nominal pore size are used to process water-sediment samples for the determination of organic compounds. Field application is mostly for filtration of water-sediment samples to isolate dissolved and suspended phases, allowing separate determinations of organic compounds in each phase. For hydrophobic organic compounds that partition to sediment organic matter, filtration is mainly used to concentrate suspended sediment on the filter to improve extraction efficiency and lower analytical detection limits. This process is especially useful for samples with low concentrations of suspended material where large volumes (4-40 L) can be filtered to provide a larger mass of suspended sediment than if a typical 1-L sample were extracted. For hydrophilic organic compounds, filtration is used to remove suspended material trapped by the filter because it often contains analytical interferences that are co-extracted with the compounds of interest. Filtration also is used as a method of sample preservation for organic compounds because it removes microorganisms from the sample (Ogawa and others, 1981).

Description

Glass-fiber filters are made of borosilicate glass and combine the properties of a depth filter and a microporous medium. The glass-fiber mesh retains sediment particles by entrapment within the mesh. In addition, the fiber mesh forms an approximate pore-size distribution across the surface of the filter, providing some of the characteristics of a microporous membrane. Particle-retention efficiency is determined by the fineness and length of the constituent microfiber and depth of the fiber mat. Because of the random nature of the glass-fiber mesh, the size of

the particles retained is less precise than for membrane filters. The particle-retention efficiency is expressed as the smallest particle size at which a retention level of 95 to 98 percent is attained. For this reason, the glass-fiber filters are described by a nominal particle-retention rating (in micrometers).

The surface of the glass filter is basically inert and is resistant to organic solvents used in precleaning or extracting organic compounds from sediment particles trapped by the filter. Filters without binders must be used with samples for organic analysis because acrylic resin binders can leach and contaminate samples.

Binder-free glass-fiber filters are available in a variety of standard disc sizes ranging from 13 to 293 mm in diameter. The smallest nominal pore size of glass-fiber filters currently (1995) available is 0.7 μm . Preclean the filters by baking in a muffle furnace at 450°C for at least 2 hours.

Disposable Membrane Filters

Application

Nylon membrane filters in disposable polypropylene units are used to process water-sediment samples for determination of organonitrogen herbicides by an SPE method (Sandstrom and others, 1992). This method requires that suspended particles be removed from the water-sediment sample to prevent blockage of the solid-phase column. While membrane filters generally are not suitable for organic samples, disposable nylon membrane filters are suitable in this particular case because the method uses GC/MS and SIM for determining the herbicides, and potential interferences from the plastic components of the filter units are not detected. Also, the sample volume (100 mL) is small enough that blocking of the membrane filter is not a problem. Finally, the organic compounds deter-

mined by this method have a low tendency to sorb to the surfaces of the filter unit or partition into particulate organic matter. As a result, the particulate material trapped on the filter is not needed for analysis.

NOTE: Nylon or other membrane filter units might be suitable for other organic methods, but appropriate quality-control samples such as blanks and spiked samples need to be analyzed prior to use.

Description

The membrane filters tested for use in the determination of organonitrogen herbicides in water-sediment samples by SPE are made of a thin film of nylon 25 mm in diameter and have a pore size of 0.45 μm . The membrane is contained in a disposable housing made of polypropylene, with female luer inlet and male luer outlet connections. The filter units need to be cleaned prior to use by rinsing with about 10 mL methanol.

Positive Displacement Pump Filtration System

Application

Pumping filtration systems force water through a filter by positive pressure from a pump placed between the unfiltered sample and filter unit. They can filter both large and small volumes of water that have moderate to high concentrations of suspended sediment. Pumping filtration systems that are used to process water-sediment samples for determination of organic compounds need to be constructed of materials compatible with the organic solvents used for cleaning and not cause any sorptive losses. These materials include stainless steel, aluminum, fluorinated polymers such as Teflon-FEP, glass, and nonporous ceramics (hard-fused micro-crystalline alumina). Other materials proven to be sufficiently resistant to attack by solvents used in

cleaning, so as not to introduce contaminants or cause absorptive losses, can be used. Plastics, rubber, oils, and other lubricants need to be avoided to prevent sample contamination and sorptive losses.

Description

Positive-displacement pumping systems (fig. 1 and table 1) consist of an in-line filter holder, a valveless, piston-type

metering pump, and Teflon-FEP (fluorinated ethylene propylene) tubing. The filter units are available in standard diameters ranging from 13 to 293 mm, and the size selected is determined by the sample volume and concentration of suspended sediment. The filter units are available with stainless steel, aluminum, or Teflon components, depending on the diameter of filter unit.

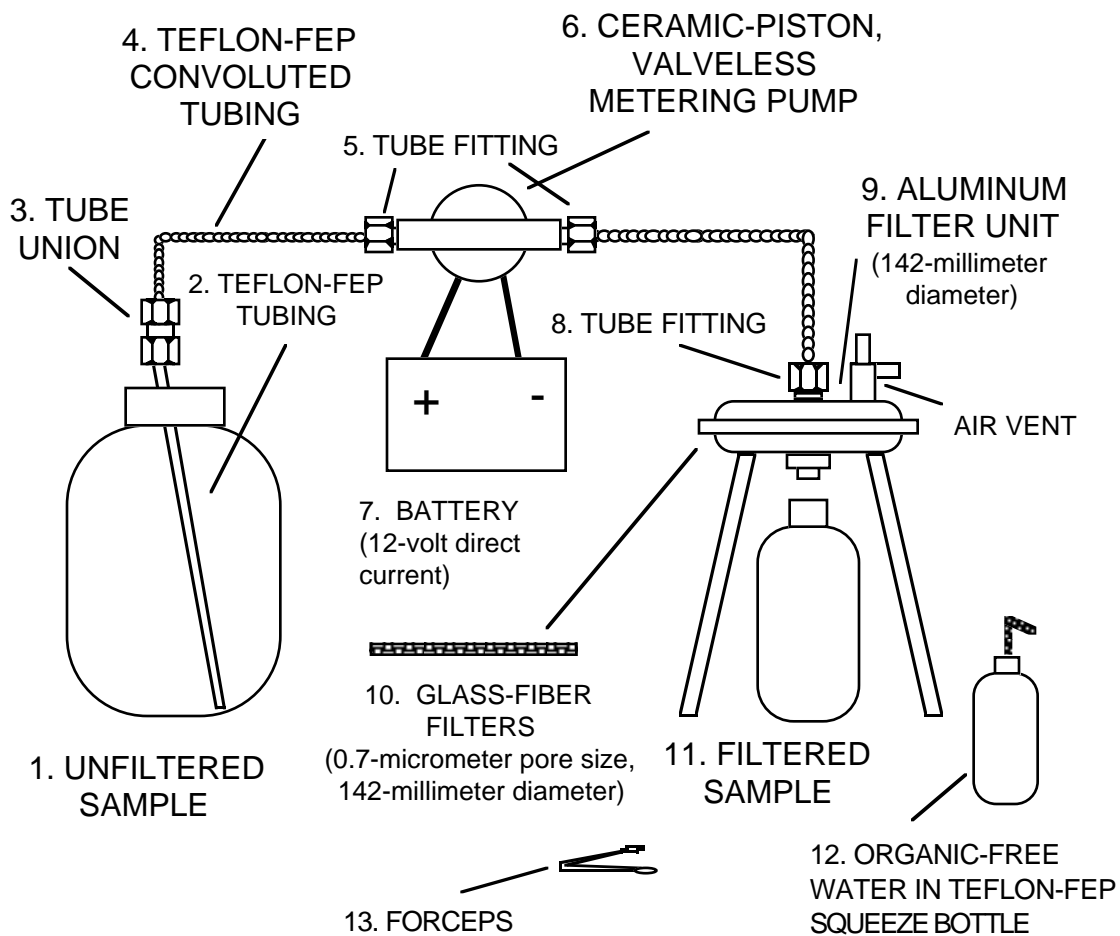


Figure 1.--A positive-displacement filtration system suitable for organic compound determinations. (Refer to table 1 for a description of numbered components.)

Table 1.--Equipment for filtration of water-sediment samples for determination of organic compounds (See fig. 1)

[mm, millimeter; mL/min, milliliters per minute; L, liter;
 μ m, micrometer; °C, degree Celsius]

Item number	Description of equipment
1	Sample container for unfiltered sample. Clean glass bottles with Teflon-FEP-lined lids, or Teflon sampling bottles are suitable.
2	Teflon-FEP tubing, 6.35-mm outside diameter.
3	Union, 6.35-mm tube (Swagelock Co., Solon, Ohio, No. SS-400-6 or equivalent).
4	Teflon-FEP convoluted tubing, 6.35-mm outside diameter (Cole-Parmer Instrument Co., Chicago, Ill., No. L-06486-02 or equivalent).
5	Tube fitting, 6.35-mm diameter tube to 6.35-mm diameter pipe thread (Swagelock Co., Solon, Ohio, No. SS-400-1-4 or equivalent).
6	Pump, ceramic-piston, valveless, with 12-volt direct current motor, capable of pumping from 0 to 500 mL/min (Fluid Metering, Inc., Oyster Bay, N.Y., Model QB-1 CSC or equivalent).
7	Battery, 12-volt direct current.
8	Tube fitting, 6.35-mm diameter tube to 9.53-mm diameter pipe thread (Swagelock Co., Solon, Ohio, No. SS-400-1-6 or equivalent).
9	In-line filter unit, aluminum, 142-mm diameter (Geotech Environmental Equipment Inc., Denver, Colo., No. 0860 or equivalent).
10	Glass-microfiber filters, binder-free, 142-mm diameter, 0.7- μ m nominal pore size (Whatman Inc., Clifton, N.J., GF/F grade, No. 1825C142 or equivalent). Note: Filters require heating at 450°C for at least 2 hours before use.
11	Filtered sample bottle, amber borosilicate glass, 1 L with Teflon-FEP-lined cap.
12	Teflon-FEP squeeze bottle for organic-free water.
13	Stainless-steel forceps for handling filters.

The valveless, piston-type metering pump consists of a pump head with a reciprocating piston driven by a 12-volt direct current (dc), variable speed motor. The pump head and all wetted parts are constructed of ceramic or Kynar-PVDF (polyvinylidene fluoride) components, which are resistant to organic solvents. Although these pumps can tolerate suspended solids in the pumped fluid, high levels of suspended solids can cause excessive demand on the pump motor. The ceramic shaft of these pumps will break if the motor exceeds 4 amperes (A). To avoid this problem, use either a 4-A dc breaker in-line with the pump, or a 115- to 12-volt converter with 4-A maximum output. A valveless,

piston-type metering pump commonly used by the U.S. Geological Survey (table 1) is capable of pumping from about 0 to 500 mL/min. A Teflon-TFE (polytetrafluoroethylene) diaphragm pump head for Masterflex pump drives, available from Cole-Parmer Instrument Company, also is satisfactory.

Connect the pump and filter unit with 6.35-mm diameter convoluted Teflon-FEP tubing. The convoluted tubing does not crimp when bent, unlike straight Teflon tubing.

The filter holders most widely used for filtration of water-sediment samples for determination of organic compounds

by the U.S. Geological Survey consists of two machined aluminum or stainless-steel plates, 142-mm diameter, held together by three locking bolts or a locking ring. The plates have Teflon, Teflon-coated silicone, or Viton o-rings set in grooves to seal the filter, and a stainless-steel support screen on the lower plate for the filter. A valve is located in the upper plate to exhaust trapped air. Threaded holes 9.53-mm diameter at the center of each plate are used to attach tubing fittings for inlet and outlet Teflon tubing.

For small sample volumes or samples with relatively low concentrations of suspended sediment, 47-mm diameter flowthrough filter units can be used. These filter units consist of two Teflon plates held together by a threaded locking ring. The bottom plate contains a Teflon support screen for the filter, and threaded holes 6.35-mm diameter at the center of each plate are used to attach fittings for inlet and outlet Teflon tubing. Wrap Teflon tape around the threads of the upper Teflon plate to prevent leaks during filtration.

Other in-line filter units, including 13- and 25-mm stainless-steel filter units, or the disposable nylon membrane filter unit used in the organonitrogen SPE method, can be connected to the pumping system using appropriate tubing-to-luer reducing fittings (see fig. 2 later in this report).

Care and Maintenance

The filter assemblies need to be thoroughly cleaned prior to use with a nonphosphate laboratory detergent (0.2 percent solution of Liquinox or equivalent). Disassemble the unit, remove any filters, and immerse in a sink or container and gently scrub with a soft brush. Rinse the components with copious amounts of tap water to remove all traces of detergent, followed by a rinse with ultrapure methanol. Clean the positive displacement pump and attached

Teflon tubing by pumping laboratory detergent through the tubing followed by rinses with tap water and methanol.

NOTE: Detergents and methanol are necessary in the cleaning procedures for trace organic contaminants. However, they might cause bias in the analyses of other organic constituents, such as dissolved organic carbon, methylene blue active substances, and certain specific surfactants. Thorough rinsing with hot tap water, or the use of separate equipment, is needed to analyze these constituents.

CAUTION: When rinsing with methanol, use a fume hood or well-ventilated area and take necessary safety precautions to prevent skin and eye contact. Use plastic trays to collect the methanol rinse, transfer to disposal containers, and dispose of the used methanol according to local regulations.

For samples containing relatively high concentrations of hydrophobic organic compounds such as polychlorinated biphenyls, the methanol rinse may not be sufficient to remove all traces of contaminants. In that case, additional rinsing with dichloromethane or hexane after the methanol rinse might be required. Information about the need for these additional rinses will need to be obtained from previous analyses of samples from the site, or field test kits and field GC or other analytical equipment.

NOTE: These solvents (dichloromethane or hexane) are not soluble in water, so they must be used for rinsing only after the equipment is dry (the methanol rinse speeds the drying procedure). After cleaning, reassemble the filtration system and cover all openings with aluminum foil for storage between uses.

PROCEDURES

A relatively clean workspace for sample filtration is required at the field site. It is important to select a location that can be protected from potential contamination from exhaust fumes and dust. Appropriate areas include a bench installed in a field vehicle or a table in a building conveniently located near the sampling site. Clean all sampling equipment before each use according to recommended procedures.

Use sampling methods and devices that are capable of collecting water samples that accurately represent the water-quality characteristics of the water body at a given time or location. Detailed descriptions of sampling methods used by the U.S. Geological Survey for obtaining depth- and width-integrated samples from streams are given in Edwards and Glysson (1988). The US-series samplers described in these documents need to be used with Teflon nozzles and glass or Teflon sample containers when sampling for anthropogenic organic compounds. A 19- or 38-L stainless-steel milk can is a suitable container for holding a depth- and width-integrated composite sample prior to filtration. Procedures for obtaining representative ground-water samples are described by Barcelona and others (1985) and Hardy and others (1989). The in-line filter unit can be connected to the outlet line of a down-hole ground-water sample pump in this case, provided the pump material is suitable for sampling organic compounds.

General Filtration Procedures for Organic Compounds

Cover the bench or table with a sheet of aluminum foil to make a clean workspace. Place the sample to be filtered, filter unit, pump system, pre-cleaned filters (baked at 450°C), forceps, and sample bottles (fig. 1) on the clean workspace, and remove the protective aluminum-foil wrapping from the

openings of the clean equipment. Pre-cleaned filters are stored in glass jars or wrapped in aluminum foil to prevent contamination. Wear disposable latex gloves to avoid contamination of the clean filter unit. Remove the upper plate of the filter unit and place a pre-cleaned glass-fiber filter onto the lower plate of the filter unit, using stainless-steel forceps to manipulate the filter. Be careful not to puncture or tear the filter, and avoid touching the filter with fingers. Wet the filter unit with a few drops of organic-free water contained in a Teflon squeeze bottle to help keep the filter in place as the unit is reassembled. Replace the upper plate of the filter unit, gently tightening the hold-down clamps. Add 10 to 20 mL of organic-free water through the upper filter plate inlet opening to completely wet the filter, and firmly tighten the clamps. If the filter is dry, it may tear when the unit is tightened. Connect the pump tubing to the inlet of the filter unit.

Remove the protective cover from the inlet line of the filter unit, and place it into the composite sample. Place a container under the outlet of the filter unit, and turn on the pump to rinse about 125 mL through the lines to remove any methanol that remains from cleaning procedures (if using a 142-mm diameter filter unit). An 8-ounce plastic bottle can be used as an approximate measure. Turn off pump and discard rinse. Then begin sample filtration by placing an appropriate sample container under the outlet of the filter unit and turning on pump. Collect sufficient sample required for each analysis. Do not completely fill bottles; leave at least 2- to 3-cm headspace to allow addition of matrix spike standards (if required) and to prevent sample loss by expansion or from spilling when the bottles are opened at the laboratory.

If the filter becomes clogged before the required volume of sample has been collected, replace it with a new filter and continue the filtration. Allow the next 125 mL (collected in a waste container

such as 8-ounce plastic bottle) to rinse the filter unit to remove any sediment particles that may have moved below the filter during the replacement procedure. This volume of sample rinsed through the filter must be measured and recorded if the sediment collected on the filter is to be analyzed for organic contaminants.

NOTE: The filters cannot be back-flushed because the filter units do not have upper screens.

When filtration is completed or the filter becomes blocked, remove as much water as possible from the inside of the filtration unit by pumping air through the lines or using a syringe to apply pressure to the inlet of the filter unit. This procedure prevents spilling of a water-sediment slurry when the filter unit is disassembled and the top plate is removed. When all water from the filter unit has passed through the filter, release the clamps and remove the top plate of the filter unit. Using stainless-steel forceps, carefully fold the filter in quarters and remove from the filter unit; discard or transfer the filter to a clean glass jar if suspended sediment constituents are to be analyzed.

If the concentration of anthropogenic organic compounds in the suspended sediment phase is to be determined, the volume of filtered water needs to be accurately measured (± 1 mL) at the field site by using a balance or measuring cylinder.

Specific Filtration Procedures for Organonitrogen Herbicides

Cover the bench or table with a sheet of aluminum foil to make a clean workspace. Assemble the sample to be filtered, disposable filter unit, pump system, and sample bottles (fig. 2 and table 2) on the clean workspace, and remove any protective aluminum-foil wrapping from openings of the clean equipment. Wear disposable latex gloves to avoid contamination of the

pump tubing. Place inlet line from the pump into the sample to be filtered. The disposable filter units do not have air vents so air needs to be removed from the lines prior to attaching the filter unit. Turn on pump and pump a few milliliters of sample to remove air from the pump and tubing. With the pump turned on, connect the tubing from the pump to the inlet of the filter unit, and begin collecting filtrate in a glass sample container. The filters have been pre-cleaned with methanol (refer to p. 4), so a filter rinse is not required. Do not completely fill the sample container; leave a 2- to 3-cm headspace. If the filter becomes blocked before sufficient sample has been filtered, the filter unit can be replaced and filtration continued until at least 100 mL has been collected. When completed, discard filter units and rinse pump and tubing with detergent, followed by tap water and ultrapure methanol.

Equipment Blanks

All sampling programs need to have a field quality-assurance protocol that includes field blanks, as well as field spikes and replicates (Sandstrom, 1990). This is especially important in the analysis of organic compounds at low concentrations because of potential bias from contamination or sorption. A field blank is a sample of organic-free water processed through the entire sampling and analytical procedure. This process includes pumping through the filtration units and filters discussed in this report. Evaluation of the results of such equipment blanks will help determine if the cleaning procedures are sufficient, or if sorption and cross contamination are problems.

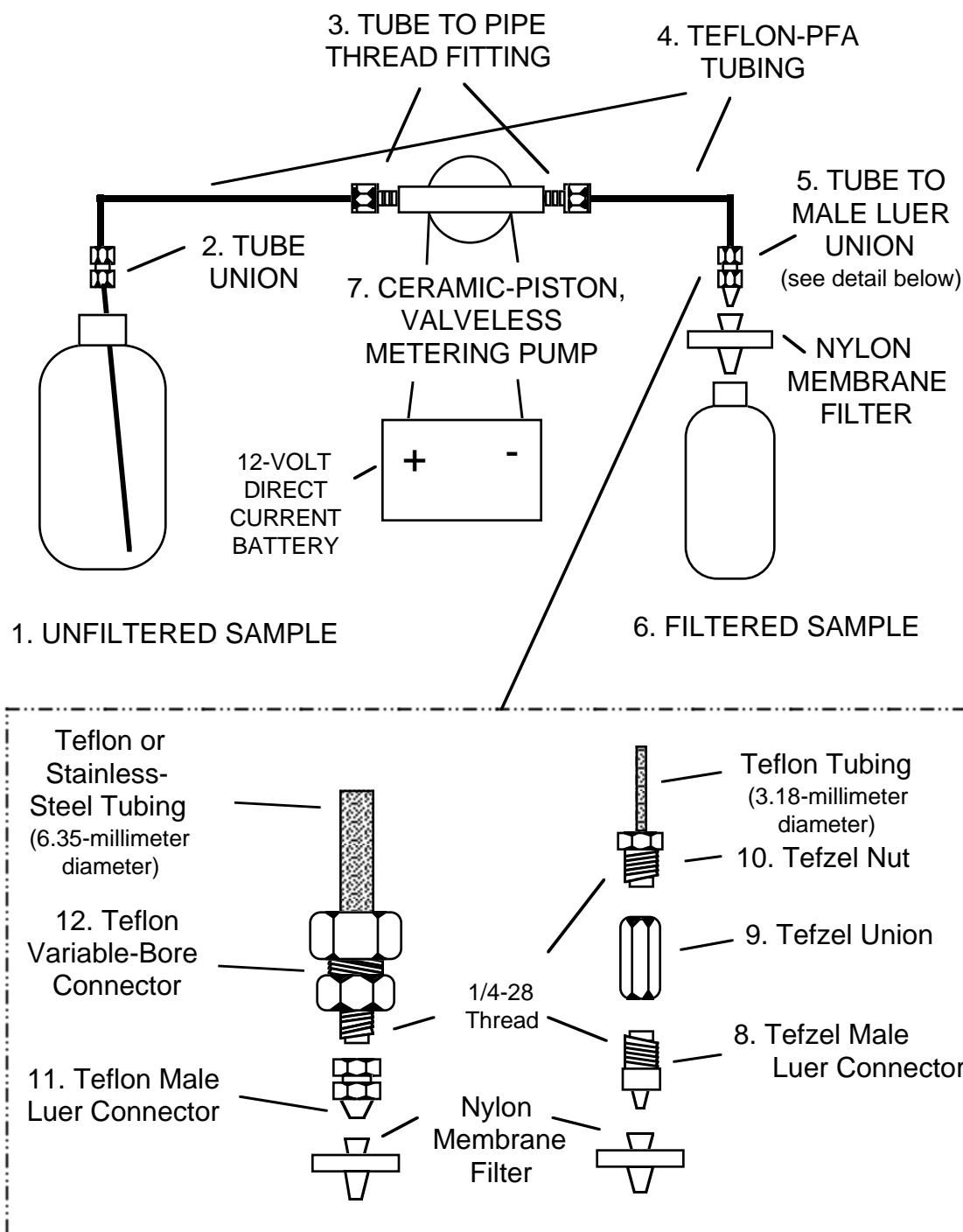


Figure 2.--Filtration system for preparation of water-sediment samples for determination of organonitrogen herbicides by solid-phase extraction. Detail gives examples of various fittings available for connecting different size tubing to filters. (Refer to table 2 for a description of numbered components.)

Table 2.--Optional equipment using disposable in-line filter holders for filtration of water-sediment samples for determination of organonitrogen herbicides by solid-phase extraction
(Sandstrom and others, 1992) (See fig. 2.)

[mm, millimeter; mL, milliliter; mL/min, milliliters per minute]

Item number	Description of equipment
1	Sample container for unfiltered sample. Clean glass bottles with Teflon-FEP-lined lids or Teflon sampling bottles are suitable.
2	Union, 3.18-mm tube (Swagelock Co., Solon, Ohio, No. SS-200-6 or equivalent).
3	Tube fitting, 3.18-mm diameter tube to 6.35-mm diameter pipe thread (Swagelock Co., Solon, Ohio, No. SS-200-1-4 or equivalent).
4	Teflon-PFA tubing, 3.18-mm outside diameter (Cole-Parmer Instrument Co., Chicago, Ill., No. CL-06375-01 or equivalent).
5	Tube to male union for either 6.35- or 3.18-mm diameter tubing.
6	Filtered sample container, amber borosilicate glass, 125 mL with Teflon-FEP lined cap.
7	Pump, ceramic-piston, valveless, with 12-volt direct current motor, capable of pumping from 0 to 500 mL/min (Fluid Metering, Inc., Oyster Bay, N.Y., Model QB-1 CSC or equivalent).
8	Tefzel male luer to 1/4-28 thread connector (Upchurch Scientific, Oak Harbor, Wash., No. P-625 or equivalent).
9	Tefzel union, 1/4-28 thread connector (Upchurch Scientific, Oak Harbor, Wash., No. P-631 or equivalent).
10	Tefzel nut, 3.18-mm tubing to 1/4-28 thread connector (Upchurch Scientific, Oak Harbor, Wash., No. P-315 or equivalent).
11	Male luer fitting, Teflon (Omnifit USA, New York, N.Y., No. 2502 or equivalent).
12	Universal connector, Teflon, for tube up to 11-mm outside diameter (Omnifit USA, New York, N.Y., No. 1008 or equivalent).

SUMMARY

An on-site filtration procedure for the determination of organic compounds in surface and ground water is described. The procedures and equipment are used in USGS water-quality studies. Previous procedures required filtration in the laboratory and used membrane filters that became clogged before the necessary volume of sample could be filtered. This was not a problem in early water-quality studies designed for regulatory purposes that focused on whole-water determinations. However, more recent studies have focused on dissolved or

particulate phase determinations, or have needed filtration as a prerequisite for the extraction procedure.

The on-site procedure has several advantages compared to previous procedures. Larger sample volumes can be filtered because the depth-filters have higher sample loading capacity than membrane filters. On-site filtration also might preserve the integrity of the samples during shipment to the laboratory because filtration removes microorganisms that can alter the sample. Filtration improves analytical determinations by removing co-extracted interferences.

Glass-fiber filters are used to process water-sediment samples for the determination of organic compounds. The borosilicate glass filters are generally inert and resistant to organic solvents used in precleaning or extraction procedures. The smallest nominal pore size of glass-fiber filters currently (1995) available is 0.7 μm . The size of the particles retained is less precise than for membrane filters because of the random nature of the glass-fiber mesh.

Nylon membrane filters contained in disposable polypropylene housing units, commonly 25-mm diameter and 0.45- μm pore size, are used to process water-sediment samples for determination of organonitrogen herbicides by a GC/MS SIM method. Membrane filters are suitable in this case because potential interferences from the plastic components of the filter units are not detected using the GC/MS SIM method, sample volumes are relatively small, and the compounds determined do not sorb to the filter unit.

Positive-displacement pumping systems used for determination of organic compounds consist of an in-line filter holder, a valveless, piston-type metering pump, and Teflon-FEP tubing. All equipment is composed of materials that neither introduce contaminants nor cause sorptive losses. These materials include stainless steel, aluminum, fluo-rinated polymers such as Teflon-FEP, glass, and nonporous ceramics (hard-fused microcrystalline alumina). All equipment is thoroughly cleaned with a nonphosphate laboratory detergent and rinsed with tap water to remove all traces of detergent, followed by a rinse with ultrapure methanol.

A relatively clean workspace and appropriate care to avoid contaminating the samples is required during filtration. Aluminum foil is used to cover open parts of the filtration equipment, latex gloves are used to avoid contamination of the filter unit, and forceps are used to handle the filters. If the filter becomes

clogged during filtration before the required volume of sample has been collected, the filter is replaced with a new filter and the filtration continued. These filter units cannot be backflushed because they do not have upper screens.

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